

Appln. No. 10/522,672  
Rule 132 Declaration

DOCKET NO: 264704US0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :  
STENZEL, OLEG, ET AL : ART UNIT: 1797  
SERIAL NO: 10/522,672 :  
FILED: AUGUST 16, 2005 : EXAMINER: SMITH, J.  
FOR: PRECIPITATED SILICA WITH :  
HIGH SURFACE AREA :

DECLARATION UNDER 37 CFR 1.132

COMMISSIONER FOR PATENTS  
P. O. BOX 1450  
ALEXANDRIA, VIRGINIA, 22313-1450

SIR:

Now comes ANDRE' WEHMEIER who deposes and says that:

- (1) I am an inventor of the above-identified invention.
- (2) Since 1998 I have been employed by EVONIK as a researcher engaged in the study of PRECIPITATED SILICAS AND RUBBERREINFORCEMENT.
- (3) I have considered the Office Action of March 28, 2008 and the Uhrlandt et al U. S. Patent 6,180,076 cited therein.
- (4) That in order to demonstrate that the claimed precipitated silica of the present invention, which has the property characteristics set forth in the product claims, results in a tire product of superior high speed characteristics in comparison to a tire product prepared from the

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precipitated silica, as a filler, described in Example 4 of Uhrlandt et al, the following comparative evidence has been obtained and hereby presented.

#### Example 5 (Invention)

A 29.2 liter amount of water and 0.01 kg of water glass (density: 1.352 kg/l, 27.6 %  $\text{SiO}_2$ , 8.20 %  $\text{Na}_2\text{O}$ ) are introduced into a reactor made of high quality steel, which has a propeller stirring gear, and is equipped with a double shell for heating purposes. While stirring the aqueous water glass solution vigorously at 66° C for 66 minutes, 7.90 l/hr of water glass and about 1.90 l/hr sulfuric acid (density: 1.40 kg/l, 50.6 %  $\text{H}_2\text{SO}_4$ ) are added to the aqueous solution. The sulfuric acid is metered into the solution in such a way that the pH of 10.0 (measured at room temperature) is established in the reaction medium. Then the addition of water glass is stopped and the sulfuric acid is added until a pH of 3.0 (measured at room temperature) is achieved. The suspension that is obtained in this manner is filtered and rinsed with water. The silica slurry has a 17 % solids content and is spray-dried. The pulverulent product that is thus obtained has a BET surface area of 283  $\text{m}^2/\text{g}$ , a CTAB surface area of 231  $\text{m}^2/\text{g}$ , DBP absorption of 293 g/(100 g), a Sears number  $V_2$  of 29.7 ml/(5 g) and a conductivity of 320  $\mu\text{S}/\text{cm}$ .

#### Example 6 (Comparative)

The precipitated silica of Example 4 of Uhrlandt et al (EP 0901986) was selected as the filler for the preparation of a rubber compound mixture.

The formulation used for the rubber mixtures of the two examples is that disclosed in Example 4, Tables 5 and 6 of the present specification. The silica of Example 4 of Uhrlandt et al (BET surface area of 185  $\text{m}^2/\text{g}$ , a CTAB surface area of 163  $\text{m}^2/\text{g}$ , a DHP index of 269 ml/100 g

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and a Sears index of 17.0) was modified with 6.4 phr Si 69 and 1.51 phr sulfur. In order to take into consideration the higher surface area of the present invention of Example 5 above, the quantity of silane was increased to 8 phr and the quantity of sulfur was reduced to 1.33. A sulfur correction is necessary for a correspondence of the sulfur content of the silane (H. D. Luginsland, J. Frohlich, A. Wehmeier, paper No. 59, April meeting of the ACS in 2001).

The table below shows the results of the rubber-technical tests of Examples 5 and 6.

		Example 4 of EP0901986	Example 5 of the invention
Silica	phr	80	80
Silane	phr	6,4	8,0
<i>ML(1+4) at 100 °C 3<sup>rd</sup> stage</i>	MU	66	104
Delta torque	dNm	16,8	23,5
t 10 %	min	1,7	0,8
t 90 %	min	6,5	15,3
<i>vulcanization time</i>	min	20	20
<i>Tensile strength (6 rings)</i>	MPa	13,0	11,8
Modulus 100 %	MPa	2,0	2,3
Modulus 300 %	MPa	10,1	9,9
Elongation at break	%	350	335
<i>Shore-A-hardness</i>	SH	66	72
<i>DIN-Abrasion, 10 N</i>	mm <sup>3</sup>	86	73
<i>Ball-Rebound, 60 °C</i>	%	63,8	59,7
E*, 0 °C	MPa	19,8	48,1
E*, 60 °C	MPa	8,0	14,9
tan δ, 0 °C	—	0,463	0,408
tan δ, 60 °C	--	0,110	0,115
considered sum of peaks	--	84	54
Peak area (Topo)	%	3,8	1,9

5) The dynamic modulus tests at  $E^*$  ( $0^\circ \text{ C}$  and  $60^\circ \text{ C}$ ) in the table above show increased values for the rubber product of Example 5 in comparison to the corresponding values of Example 6. These greater values of the dynamic modulus tests correspond to higher rigidities of the present rubber than the rubber of Example 6 (comparative). The higher rubber rigidity is especially important for high speed passenger vehicles and motor cycle tires, as it is evidence of improved traction under dry conditions and higher cornering stability. The higher CTAB values of the present precipitated silica lead to reduced attrition, but maintains a rolling resistance of the tire product comparable to that which is prepared from the precipitated silica of Example 4 of Uhrlandt et al. This effect was unexpected, because one of skill in the art would expect that upon an increase in the CTAB values, an increase in rolling resistance would be observed. This unfavorable effect, however, has not been observed using the precipitated silica of the present invention. In spite of the silica with higher CTAB surface area of the silica of the present invention, the  $\tan \delta$  ( $60^\circ \text{ C}$ ) values are, favorably, almost unchanged in comparison to the silica of Example 4 of the EP '986 reference. The improvement in road abrasion can also be achieved when the high surface area silicas of the present invention are used in natural rubber mixtures, such as those used in truck tire tread mixtures. In particular, in combination with a high surface area, high structure carbon black such as N 121, having excellent road abrasion resistance, can be achieved in truck tires. Especially in this area of improvement in the cut and chip and chunking behavior is of substantial interest and can be implemented by use of the high surface area silicas of the present invention.

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(6) The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

(7) Further deponent says not.

31.07.2008

(Date)

